[Title of Invention]

METHOD OF MANUFACTURING Ti-CONTAINING STEEL TO MAKE TiN INCLUSION FINE

[Abstract]

[Problem to be Solved]

To provide a method of manufacturing a Ti-containing steel with TiN system inclusions, which deteriorates the steel in fatigue duration, and which have a length of not more than 10 μm.

[Solution]

The method of manufacturing the Ti-containing steel, in which TiN system inclusions are made fine, includes melting raw materials for the Ti-containing steel which contain no recirculating materials in a vacuum induction furnace, preparing an electrode from a casting of the Ti-containing steel after the vacuum melting, and remelting the electrode by a vacuum arc melting method.

[CLAIMS]

[Claim 1]

A method of manufacturing a Ti-containing steel, in which steel TiN system inclusions are made fine, characterized by melting raw materials for the Ti-containing steel which raw materials contain no TiN system inclusions in a vacuum induction furnace, preparing an electrode from a casting of the Ti-containing steel after the vacuum melting, and remelting the electrode by a vacuum arc melting method.

[Claim 2]

The method of manufacturing a Ti-containing steel, in which steel TiN system inclusions are made fine, characterized by melting raw materials for the Ti-containing steel which raw materials contain no TiN system inclusions in a vacuum induction furnace; preparing an electrode from a casting of the Ti-containing steel after the vacuum melting; remelting the electrode by a vacuum electroslag melting method; preparing an electrode from a remelted material produced by the vacuum electroslag melting method; and further remelting the second electrode by a vacuum arc melting method.

[Claim 3]

The method according to claim 1 or 2, characterized by that the remelting by the vacuum arc melting method is carried out at a molten metal rising rate of not more than 0.4 cm/min.

[Detailed Description of the Invention]

[0001]

[Field of the Invention]

The present invention relates to a method of manufacturing a Ti-containing steel, such as maraging steels, to make TiN type inclusions contained therein fine.

[0002]

[Technical Background of the Invention]

Since maraging steels, which are one type of Ti-containing steels, have high hardness/strength and high hot/cold fatigue properties, they are used for applications to various dies, rockets using a solid fuel, ultracentrifuges, torque transmission shafts, heavy-duty gears and so on. In general, maraging steels are manufactured by a two step melting method in which a material manufactured by melting in a vacuum induction melting furnace is again melted by a vacuum arc remelting method to cast the melt.

[0003]

The two step melting method involves, first, (1) adjusting alloying components, and (2) reducing impurities such as C, N, H and O with use of a vacuum induction furnace, and as a second stage, further reducing C, N, H, O, etc. and reducing the segregation by buildup solidification by the vacuum arc remelting. Although the maraging steels manufactured by the two step melting method are used for various applications mentioned above because of their high hardness and strength, excellent cleanability and high fatigue characteristics, since articles required to be improved in ultrahigh fatigue properties of not less than 10^7 times tends to be ruptured from a rupture initiating point of TiN type inclusions having a size of about 10 μm, further improvements in fatigue characteristics are required, and reducing and making TiN type inclusions fine by vacuum arc remelting (for example) are needed.

[0004]

[Problems to be Solved by the Invention]

It is an object of the present invention to provide a method of manufacturing a Ti-containing steel which contains TiN type inclusions having a maximum length of not more than 10 μm.

[0005]

[Means for Solving the Problems]

As a result of extensive studies on methods of making TiN type inclusions in Ti-containing steels fine, chemical compositions and manufacturing processes thereof to solve the problems mentioned above, the present inventor found that the TiN type inclusions having a length of more than 10 μm shorten the anti-fatigue life, that a vacuum arc remelting cannot remove the TiN type inclusions contained in raw materials, that a method of making the TiN type inclusions fine involves making the amount of the TiN type inclusions small, and that remelting by a vacuum electroslag melting method can reduce the TiN type inclusions.

[0006]

The present inventor found also that making small the amount of TiN type inclusions in raw materials for a Ti-containing steel can reduce the TiN type inclusions in the manufactured Ti-containing steel, that for making less the amount of TiN type inclusions in raw materials for a Ti-containing steel, it is desirable that the raw materials be carefully selected and do not contain return materials, that preventing the aggregation of TiN type inclusions during melting can make the TiN type inclusions small, and that in the case of melting by a vacuum arc remelting method, a high melting rate makes TiN type inclusions large. These findings have led to the present invention.

[0007]

That is, for solving the problems described above, the method of manufacturing a Ti-containing steel to make TiN type inclusions fine according to the present invention involves melting raw materials for a Ti-containing steel which materials do not contain TiN type inclusions (desirably do not contain return materials) in a vacuum induction furnace, casting the melt to manufacture a Ti-containing steel, and remelting the Ti-containing steel as an electrode in a vacuum arc melting method.

[0008]

Further, for solving the problems described above, the method of manufacturing a Ti-containing steel to make TiN type inclusions fine according to the present invention involves melting raw materials for a Ti-containing steel which materials do not contain TiN type inclusions (desirably do not contain return materials) in a vacuum induction furnace, casting the melt to manufacture a Ti-containing steel, remelting the Ti-containing steel as an electrode in a vacuum electroslag melting method, and further remelting as an electrode the remelted material, which has been melted by the vacuum electroslag melting method, by a vacuum arc melting method.

[0009]

Further, for solving the problems described above, the method of manufacturing a Ti-containing steel to make TiN type inclusions fine according to the present invention involves that the melting by the vacuum arc remelting method is carried out at a molten metal rising rate of not more than 0.4 cm/min.

[0010]

[Operation]

In the method of manufacturing a Ti-containing steel to make TiN type inclusions fine, since raw materials for the Ti-containing steel which materials do not contain TiN type inclusions are melted in a vacuum induction furnace, a Ti-containing steel containing a small amount of TiN type inclusions can be manufactured and if the Ti-containing steel is remelted as an electrode by a vacuum arc melting method, a Ti-containing steel containing a small amount of TiN type inclusions, further of a small shape, can be manufactured. Further, since the Ti-containing steel is melted in a vacuum induction furnace and cast to manufacture a Ti-containing steel containing a small amount of TiN type inclusions, and the Ti-containing steel is remelted as an electrode by a vacuum electroslag melting method, the TiN type inclusions are captured in the slag without being contaminated with N_2 in the air, enabling manufacture of a Ti-containing steel containing a further smaller amount of and smaller TiN type inclusions.

[0011]

An electrode and the like manufactured by remelting by a vacuum electroslag melting method are remelted by a vacuum arc melting method, thereby enabling to reduce impurities such as C, N, H and O, fining TiN type inclusions and reduce the segregation. Further, when an electrode manufactured by remelting by a vacuum electroslag melting method is remelted by a vacuum arc melting method, by making the melting rate as low as possible (desirably, the molten metal rising rate is not more than 0.4 cm/min), the pool becomes small and shallow, so since the aggregating time of TiN type inclusions in the pool becomes short, whereby the TiN type inclusions can be made fine.

[0012]

[Embodiments of the Invention]

Then, the present invention will be further in detail described. The Ti-containing steel containing TiN type inclusions made fine of the present invention refers to a maraging steel (a steel composed of C: 0.010% or less, Si: 0.05% or less, Mn: 0.05% or less, P: 0.06% or less, S: 0.006% or less, Ni: 16% to 26%, and Ti: 0.1% to 2.0%; optionally containing one or a mixture of two or more of Co: 5% to 16%, Mo: 2% to 10%, and Al: 0.03% to 0.4%; and containing Fe as the remainder and inevitable impurities), JIS SUH660 (a steel composed of C: 0.08% or less, Si: 1.00% or less, Mn: 2.00% or less, P: 0.40% or less, S: 0.030% or less, Ni: 24% to 27%, Cr: 13.50% to 16.00%, Mo: 1.00% to 1.50%, V: 0.10% to 0.50%, Ti: 1.90% to 2.35%, Al: 0.35% or less, and B: 0.001% to 0.010%; and containing Fe as the remainder and inevitable impurities), PH stainless steel and the like.

[0013]

Raw materials for the Ti-containing steel melted in a vacuum induction furnace according to the present invention desirably do not contain TiN type inclusions, that is, all the materials are desirably composed of virgin materials. This is because if return materials are contained in raw materials for a Ti-containing steel, more TiN type inclusions are contained in a manufactured Ti-containing steel, resulting in larger TiN type inclusions.

[0014]

The vacuum induction furnace used in the present invention may be one having an ordinary structure or a special structure as long as it is an induction furnace which can melt under vacuum. Further, the vacuum electroslag melting method used in the present invention is an electroslag melting method which melts under vacuum, and is not especially limited as long as it can capture TiN type inclusions

in a molten slag, but materials of a molten slag may be composed of, for example, CaF_2: 70% and Al_2O_3: 30%. The melting rate is not especially limited, but the melting is preferably carried out at a uniform rate.

[0015]

The vacuum arc remelting method used in the present invention has the purposes of reducing impurities such as C, N, H and O, fining TiN type inclusions and reducing the segregation, and is a method in which a remelted material having been melted in a vacuum electroslag melting method is remelted as an electrode by arc in a water-cooled copper mold under vacuum. In the vacuum arc remelting method, if the diameter of the water-cooled copper mold is large and the melting rate is high, the pool of the molten metal becomes large, TiN type inclusions aggregate and become large, and the segregation also becomes large, so the diameter of a water-cooled copper mold is preferably, for example, not more than 30 cm and the molten metal rising rate is preferably slower than 0.4 cm/min.

[0016]

Then, examples of the present invention will be described.

[Examples]

Example 1

Pure titanium, pure nickel, ferromolybdenum, pure cobalt, pure aluminum and electrolytic iron, as raw materials for Ti-containing steels to make respective steels having their component compositions of subexamples Nos.1 and 2 of the present invention in Table 1, were melted in a vacuum induction furnace (VIF) for respective melting times described in subexamples Nos.1 and 2 in Table 2, and cast to manufacture respective ingots. The each ingot was melted as an electrode by a vacuum arc melting method (VAR) under the vacuum degree

and melting rate described in Table 2, and cast to manufacture an ingot. The ingot was forged and hot rolled to manufacture a hot coil of 3.5 mm in thickness. The coil was cut and measured for the size of TiN type inclusions in the cross section. The results are shown in subexamples Nos.1 and 2 of the present invention in Table 3.

[0017]

Example 2

Pure titanium, pure nickel, ferromolybdenum, pure cobalt, pure aluminum and electrolytic iron, as raw materials for Ti-containing steels to make respective steels having their component compositions of subexamples Nos.3 to 5 of the present invention in Table 1, were melted in a vacuum induction furnace (VIF) for respective melting times described in subexamples Nos.3 to 5 in Table 2, and cast to manufacture respective ingots. The each ingot was melted by a vacuum electroslag melting method (vacuum ESR) under the vacuum degree and melting rate described in Table 2, and cast to manufacture an electrode, and the electrode was melted by a vacuum arc melting method (VAR) under the vacuum degree and melting rate described in Table 2, and cast to manufacture an ingot. The ingot was forged and hot rolled to manufacture a hot coil of 3.5 mm in thickness. The coil was cut and measured for the size of TiN type inclusions in the cross section. The results are shown in subexamples Nos.3 to 5 of the present invention in Table 3.

[0018]

[Table 1]

[0019]

[Table 2]

[0020]

Comparative Example 1

The same raw materials for a Ti-containing steel as in Example 1 were melted in a vacuum induction furnace (VIF) for a melting time described in comparative subexample No.1 in Table 2, and cast to manufacture an ingot. The ingot was melted as an electrode in a vacuum arc furnace under the vacuum degree and melting rate (220 kg/h) described in comparative subexample No.1 in Table 2, and cast to manufacture an ingot. The ingot was forged and hot rolled to manufacture a hot coil of 3.5 mm in thickness. The coil was cut and measured for the maximum length of TiN type inclusions in the cross section. The result is shown in comparative subexample No.1 in Table 3.

[0021]

Comparative Example 2

82%, 88% and 65% (comparative subexamples Nos.2, 3 and 4, respectively) of a return material containing Ni, Mo and Fe and 18%, 12% and 35% (comparative subexamples Nos.2, 3 and 4, respectively) of the total of pure titanium, pure nickel, ferromolybdenum, pure cobalt, pure aluminum and electrolytic iron, as raw materials for Ti-containing steels to make respective steels having their component compositions of comparative subexamples Nos.2 to 4 in Table 1, were melted in a vacuum induction furnace (VIF) for respective melting times described in comparative subexamples Nos.2 to 4 in Table 2, and cast to manufacture respective ingots. The each ingot was melted as an electrode by a vacuum arc melting method (VAR) under the vacuum degree and melting rate described in Table 2, and cast to manufacture an ingot. A similar hot coil of 3.5 mm in thickness was manufactured using the ingot by the same method as in Example 1. The coil was cut and measured for the maximum length of TiN type inclusions in the

cross section. The results are shown in comparative subexamples Nos.2 to 4 in Table 3.

[0022]

[Table 3]

[0023]

These results reveal that the maximum lengths of TiN type inclusions of subexamples of the present invention are 6.8 μm to 9.8 μm and any of them is not more than 10 μm. By contrast, the maximum lengths of TiN type inclusions of comparative subexamples are 12.5 μm to 15.0 μm and any of them exceeds 10 μm. The molten metal rising rate lower than 0.4 cm/min in VAR provides TiN type inclusions having a shorter maximum length than that higher than 0.4 cm/min.

[0024]

[Advantages]

The method of manufacturing a Ti-containing steel whose TiN type inclusions are made fine according to the present invention can exhibit an excellent advantage of making the maximum length of the TiN type inclusions in the Ti-containing steel to be not more than 10 μm by the constitution described above.

comparative specimens

Invention Specimens

[Table 1]

	No.	C	Si	Mn	S	Ni	Mo	Co	Ti	Al	N
本発明例	1	0.003	0.03	0.03	0.001	18.55	4.73	8.85	0.44	0.11	0.0009
	2	0.002	0.03	0.03	0.001	18.58	4.75	8.85	0.45	0.11	0.0010
	3	0.002	0.05	0.01	0.001	18.66	4.76	8.89	0.46	0.15	0.0010
	4	0.002	0.03	0.01	0.001	18.73	4.75	8.82	0.45	0.11	0.0009
	5	0.001	0.04	0.01	0.001	18.66	4.72	8.90	0.45	0.12	0.0012
比較例	1	0.002	0.03	0.01	0.001	18.69	4.77	8.80	0.45	0.10	0.0008
	2	0.007	0.04	0.01	0.001	18.43	4.74	8.74	0.45	0.12	0.0007
	3	0.004	0.06	0.03	0.001	18.33	4.73	8.80	0.43	0.10	0.0007
	4	0.008	0.10	0.04	0.001	18.36	4.90	8.80	0.50	0.12	0.0020

melting time

recirculation rate of raw material

VIF melting conditions

mold diameter

Vacuum ESR conditions　electroslag melting

melting rate

vacuum degree

vacuum arc remelting

【0019】
【表2】[Table 2]

VAR conditions

	No.	VIF溶解条件 原料リターン率	VIF溶解条件 溶解時間	真空ESR条件 真空度 Torr	真空ESR条件 溶解速度	VAR条件 鋳型径	VAR条件 真空度 Torr	VAR条件 溶解速度	VAR条件 湯上がり速度
本発明例	1	0％	175分	——	——	φ340	0.004	１４１ kg/Hr	0.32 cm/min
	2	0％	175分	——	——	φ340	0.004	２０２ kg/Hr	0.48 cm/min
	3	0％	170分	150	256kg/Hr	φ460	0.004	２１５ kg/Hr	0.27 cm/min
	4	0％	170分	150	255kg/Hr	φ340	0.004	２２５ kg/Hr	0.52 cm/min
	5	0％	170分	150	257kg/H	φ340	0.004	２９０ kg/Hr	0.67 cm/min
比較例	1	0％	165分	——	——	φ340	0.004	２２０ kg/Hr	0.50 cm/min
	2	８２％	185分	——	——	φ340	0.002	２０５ kg/Hr	0.47 cm/min
	3	８８％	180分	——	——	φ340	0.003	２０６ kg/Hr	0.47 cm/min
	4	６５％	180分	——	——	φ460	0.004	２９０ kg/Hr	0.38 cm/min

molten metal rising rate

Invention specimens

Comparative specimens

min

ＶＡＲ条件の湯上がり速度は、鋳型内での溶融金属面の上昇速度である。

【００２０】比較例１
実施例１と同様な含Ｔｉ鋼用原材料を真空誘導溶解炉（ＶＩＦ）で下記表２の比較例 No.１に記載したような溶解時間で溶解し、鋳造してインゴットを製造した。このインゴットを電極とし、真空アーク炉で下記表２の比較例 No.１に記載したような真空度、溶解速度（２２０ｋｇ／Ｈｒ）で溶解、鋳造してインゴットを製造した。このインゴットを鍛造後熱間圧延して厚さ３．５ｍｍホットコイルを製造した。このコイルを切断して断面のＴｉＮ系介在物の最大長さを測定した結果を下記表３の比較例 No.１に示した。

【００２１】比較例２
Ｎｉ、Ｍｏ、Ｆｅを含有するリターン材：８２%、８８%及び６５%（比較例の No.３）、純チタン、純ニッケル、フェロモリブデン、純コバルト、純アルミを１８%（比較例の No.２）、１２%（比較例の No.３）、３５%（比較例の No.４）からなる下記表１の比較例の No.２〜４の成分組成の鋼になるような含Ｔｉ鋼用原材料を真空誘導溶解炉（ＶＩＦ）で上記表２の比較例の No.２〜４に記載したような溶解時間で溶解し、鋳造してインゴットを製造した。このインゴットを電極とし、真空アーク溶解法（ＶＡＲ）で上記表２に記載したような真空度、溶解速度で溶解、鋳造してインゴットを製造した。このインゴットを用いて実施例１と同様な方法で同様な厚さ３．５ｍｍのホットコイルを製造した。このコイルを切断して断面のＴｉＮ系介在物の最大長さを測定した結果を上記表２の比較例 No.２〜４に示した。

【００２２】
【表３】

The molten metal rising rates in the VAR conditions refers to rising rates of molten metal surfaces in the mold.

[Table 3]

	No.	溶解方法 melting method	ＴｉＮ系介在物の最大長さ maximum length of TiN inclusions
本発明例 Invention Specimens	1	ＶＩＦ→ＶＡＲ	６．８　μｍ
	2	ＶＩＦ→ＶＡＲ	９．７　μｍ
	3	ＶＩＦ→ＶＳＲ→ＶＡＲ	７．５　μｍ
	4	〃	８．６　μｍ
	5	〃	９．８　μｍ
比較例 Comparative Specimens	1	ＶＩＦ→ＶＡＲ	１２．５　μｍ
	2	〃	１５．０　μｍ
	3	〃	１２．５　μｍ
	4	〃	１５．０　μｍ

【００２３】これら結果より、本発明例のもののＴｉ系介在物の最大長さは、６．８～９．８μｍであり、何れも１０μｍ以下であった。これに対して、比較例のもののＴｉ系介在物の最大長さは、１２．５～１５．０μｍであり、何れも１０μｍを超えていた。また、ＶＡＲにおける鋳造の湯上がり速度が０．４ｃｍ／分より遅いもののほうが、０．４ｃｍ／分より早いものよりＴｉＮ＊系介在物の最大長さが短くなっていた。

【００２４】

【効果】本発明のＴｉＮ系介在物を微細にした含Ｔｉ鋼の製造方法は、上記構成にしたことによって、含Ｔｉ鋼中のＴｉＮ系介在物の最大長さを１０μｍ以下にすることができるという優れた効果を奏することができる。

~~【手続補正書】~~

~~【提出日】平成１２年１月３１日（２０００．１．３１）~~

~~【手続補正１】~~

~~【補正対象書類名】明細書~~

~~【補正対象項目名】００２０~~

~~【補正方法】変更~~

~~【補正内容】~~

~~【００２０】~~

~~【表３】~~

	No.	溶解方法	ＴｉＮ系介在物の最大長さ
本発明例	1	ＶＩＦ→ＶＡＲ	６．８　μｍ
	2	ＶＩＦ→ＶＡＲ	９．７　μｍ
	3	ＶＩＦ→真空ＥＳＲ→ＶＡＲ	７．５　μｍ
	4	〃	８．６　μｍ
	5	〃	９．８　μｍ
比較例	1	ＶＩＦ→ＶＡＲ	１２．５　μｍ
	2	〃	１５．０　μｍ
	3	〃	１２．５　μｍ
	4	〃	１５．０　μｍ

~~【手続補正書】~~

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~~【手続補正１】~~

~~【補正対象書類名】明細書~~

~~【補正対象項目名】発明の名称~~

~~【補正方法】変更~~

~~【補正内容】~~

~~【発明の名称】　ＴｉＮ系介在物を微細にする含Ｔｉ鋼の製造方法~~